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16. Abstract

Four non-destructive testing devices were used on 21 fiberglass laminated test panels prepared by boat companies and material suppliers. Each set of panels had been formulated using (1) normal layup, (2) excess catalyst, (3) deficient catalyst, (4) low temperature cure, and (5) excess water in fiberglass. The four testing devices were (1) ultrasonic inspection, (2) dielectric moisture detector, (3) neutron source device, and (4) thermography IR dector.

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FINAL REPORT

on

EXAMINATION OF BLISTERING OF FIBERGLASS REINFORCED LAMINATES

to

UNITED STATES COAST GUARD

June 26, 1987

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by

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TABLE OF CONTENTS

TABLE OF CONTENTS
(Cont'd)

APPENDIX A

LIST OF TABLES

LIST OF FIGURES

EXECUTIVE SUMMARY

Battelle researchers have conducted a preliminary investigation into methods that may be useful in determining if a fiberglass boat hull will have a tendency to blister in service. To accomplish this task, Battelle evaluated four nondestructive testing devices to determine if any have the potential for detecting flaws in hand-made fiberglass laminates. These laminates were prepared by (1) boat companies or (2) resin suppliers and were formulated as follows:

(1) normal layup
(2) excess catalyst
(3) deficient catalyst
(4) low temperature cure
(5) excess water in fiberglass.

The resultant 21 panels were subjected to the following testing devices:

(1) ultrasonic inspection
(2) dielectric moisture detector
(3) neutron source device
(4) thermography IR detector.

In addition to the above tests, Infrared Spectroscopy was used to determine the composition of both the gel coats and the laminate resins.

Of the four nondestructive instruments evaluated, ultrasonic sensing and dielectric detecting hold the most promise for revealing flaws.

The ultrasonic devices are very sensitive and appear to probe inside a laminate much as an X-ray device outlines the internal parts of a human body. The Ultrasonic output is a series of echoes that must be translated by trained personnel. This technique reveals much about the composition of a fiberglass laminate. Once standards are developed for various 'flaws', and a specific ultrasonic detector is developed for rapid scanning, these units could scan a boat hull quickly to detect serious flaws relating to blistering. This technique might also be used to detect structural flaws.

The dielectric moisture detector identifies moisture in boat hulls. It does not seem to detect other faults but it is useful for the one purpose. Since there was no way to determine what the moisture level of the laminates was at the time of test, the lowest level of moisture detection possible is unknown.

The <u>neutron bombardment technique</u> was not effective in this work. It is thought that the unit was designed for use on composite structures much thicker than the 1/4-inch laminates used in this study and, therefore, could not detect variations in composition under the conditions used in this study. Two <u>infrared scanning cameras</u> were used in the thermography detection study developed on this program. Both were able to detect some flaws in the fiberglass laminates, but it was not possible to predict what defects were seen with any confidence. One of these units could scan a boat hull quickly but until specific standards are developed, it is impossible to predict differences in defects in boat hulls.

<u>Infrared (IR) Spectroscopy</u> can easily detect resin composition of fiberglass laminates but is a destructive test and does not lend itself to predicting defects in laminates such as addressed in this program.

In conclusion, the use of ultrasonic inspection is the most promising technique investigated on this program for detecting most flaws in boat hulls. An inspection matrix listing the probable ultrasonic response which might be obtained from atypical production situations is outlined on pages 15 and 16. More research is needed to set up standards of various defects that can be used for comparison when scanning an entire hull. Special scanning heads can be developed that will scan the entire hull quickly and accurately and with a minimum of skilled personnel.

The dielectric method detects water above a certain amount but standards must be developed to determine its sensitivity.

Neutron bombardment does not appear to be a realistic test at this time.

Thermography detection may need more work to develop it (compared to other methods) and available dollars may be better spent on ultrasonics. The use of IR is destructive but is a valuable backup in determining the composition of the various materials used in laminates.

This program has been successful in determining that predictive devices are available to identify "pre-blistering defects" but much more work is needed to develop these devices into useful instruments for the Marine Industry.

FINAL REPORT

on

EXAMINATION OF BLISTERING OF FIBERGLASS REINFORCED LAMINATES

to

UNITED STATES COAST GUARD

from

BATTELLE
Columbus Division

June 26, 1987

INTRODUCTION

The blistering of gel coat laminates on fiberglass boat hulls and other composite products exposed to water has been observed for a number of years. Blistering of these laminates has been found to occur under various conditions, with different resin systems and composite materials, different catalysts, cure mechanisms, and schedules. A great number of hypotheses have been proposed to explain blister formation. However, experimental data and verification have not been readily correlated and evaluated.

The Coast Guard has taken a keen interest in these developments because of the possibility of life threatening situations that could develop if blistering resulted in a loss of structural strength of a fiberglass hull. Also, previous work on blistering of fiberglass hulls had shown that very few standards exist in the boat building industry. The Coast Guard believes that the buyer as well as the boat manufacturer would benefit if boat building standards were instituted in the United States. If standards existed, it might be possible to trace problems, such as boat blistering. As of now, there are too many variables resulting from a wide variety of products used to produce boats.

OBJECTIVES

Original Objectives

The original objectives of this program were as follows:

(1) analytically investigate the influence of cure mechanism, and extent of cure on the tendency of the boat hull to blister;

(2) determine if unreacted constituents are present;

(3) analyze for excess or deficient peroxide catalyst;

(4) look for extraneous materials;

(5) investigate binders and pigments to determine if they are in excess or have localized concentrations; and

(6) determine test procedures which may be used to predict a tendency toward blistering in a finished laminate which has not yet failed.

Modified Objectives

During a meeting with Mr. Don Ellison (COTR) on February 10, 1986, we agreed to modify the above objectives to place emphasis on determining if a technique could be developed to predict blistering in fiberglass boat hulls. These changes were outlined in a letter to Mr. Ellison dated April 30, 1986, and are as follows:

> " as discussed in Task 1 of our proposal (774-J-9693X) to you on October 16, 1985, we will want to place less emphasis on the use of microanalytical techniques such as DSC, DTA, TMA, etc. These tests will be relegated to a secondary role in this program. We will use them only as necessary in conjunction with our nondestructive tests to warrant their use as a confirmation tool. Therefore, listed below are the techniques we will use in an effort to predict failures by blistering in fiberglass boats.
>
> (1) Thermography analysis
> (2) Acoustic Emission testing
> (3) Dielectric testing
> (4) Moisture detection

> The above techniques were discussed in our initial proposal to you with the exception of moisture detection. We plan to acquire or rent a device that uses a radioactive source to detect small amounts of moisture that may be present in fiberglass composites. It is possible that the fiberglass roving or cloth may contain excess moisture due to improper storage conditions and this excess moisture could play a substantial role in blister development."

Subsequently, it was discovered that acoustic emission was not appropriate to use for nondestructive testing on fiberglass boat hulls so ultrasonic techniques were used instead. Also, as we decided in our meeting with Mr. Ellison on February 6, 1987, an analysis of the gel coat and laminate of these experimental panels would be helpful in the overall diagnosis of the defects contained in these panels.

TEST PANELS

The Coast Guard, through Mr. Ellison, contacted various boat manufacturers/resin suppliers to obtain sample panels of fiberglass reinforced plastic that would represent sections of boat hulls. Four companies responded by furnishing five different samples. These panels represented a normal layup and four abnormal production conditions.

(1) Normal layup
(2) Wet fiberglass
(3) Excess catalyst
(4) Insufficient catalyst
(5) Normal layup, low temperature cure.

These panels were identified to Battelle only with alphanumeric markings on the backside. The was done to prevent bias on the part of the researchers doing the testing. Once the testing procedures had been completed, a letter describing the results was sent to Mr. Ellison who, in turn, submitted the panel description to Battelle. Thus, all destructive and nondestructive tests performed at Battelle were performed on panels of unknown composition.

TEST METHODS

The instruments used to perform the predictive tests on the experimental panels are described in this section.

Ultrasonic Inspection of Panels

Methods including (1) direct-contact through-transmission, and (2) direct-contact pulse-echo using both single probe and dual-element probes and frequencies of 1.0 mHz, 2.25 MHz, and 5.0 MHz were evaluated. A direct-contact single-probe pulse-echo technique was selected using 1/2-inch diameter, 5 MHz transducer and a Branson Sonoray inspection system.

The inspection procedure was based upon the assumption that both panels and the inspection technique itself were being evaluated. The procedure was planned to minimize cost of inspection probably at the expense of thoroughness in defining defect types and their seriousness.

Inspection was performed by passing a transducer along a thin oil path laid on the smooth surface of each panel. Internal reflections including size, shape, and position as well as number were monitored simultaneously. These data provided the information upon which to evaluate the conditions within the panels.

The 5 MHz probe gave generally excellent penetration through the panels with one exception which is discussed later. Complete penetration was verified by occasionally loading the backside of the panel under test with a moistened finger and observing the effect of the echo from the back surface.

Sovereign Moisture Master

This instrument was obtained from:

Sovereign Chemical Industries Limited
Barrow-in-Furness
Cumbria, England LA14 4QU.

The Sovereign Moisture Master uses high frequency radio waves to measure the dielectric constant of various materials. A number of marinas are using this instrument to measure boat hulls for moisture content.

"Technically, the Sovereign Moisture Master[1] measures moisture content by directly assessing the change in capacitance due to the presence of water in the surface under test.

Since the dielectric constant for water is 75 times that of air, high sensitivity is achieved.

In practice, materials have variations in their structure, for example, in timber the cellulose structure varies greatly between adjacent points, but if a volume or area is monitored, these variations are greatly reduced. Hence, it is desirable in measurement to monitor over an area, rather than between two points. With all previous pin type probes, the calibration depended on the resistance over a specific length and to define the length, it was necessary to use spaced pin contacts. Errors due to material structure tend to be high. The Moisture Master monitors an area and is not subject to the same restriction.

Advantages Over Other Methods

The Sovereign Moisture Master provides instant readings of moisture levels. Its radio frequency field nondestructively penetrates the material surface. Its smooth scan head radiates invisible waves which plot just where the moisture is. The given reading is unaffected by salt contamination in many materials.

The Moisture Master enables you to check moisture content of any part of a boat by an infallible, non-destructive electronic method. High frequency radio waves penetrate the material being checked and identify, visually and aurally, where water is and shouldn't be--even in the dark.

---

[1] Reproduced from the Sovereign manual.

Large hull areas can be checked for moisture content simply by passing the meter head over the surface. Additionally, by presetting the instrument, an audio signal can be produced which obviates the need to continually monitor the meter dial when large surfaces are being examined. By closer inspection of suspect areas the actual point of moisture ingress can be determined. This is normally shown by the level of moisture diminishing as readings recede from the point of breakdown. Having identified the area to be repaired, the meter can again be used to ensure that prior to recoating the area is fully dry and receptive to resin treatment."

Hydrotector® Moisture Detector

This instrument, Model MC-M, was rented from the CPN Corporation, 2830 Howe Road, Martinez, California 94553. According to their literature, it has been used successfully to measure moisture content of roofs on various buildings throughout the United States.

General Operation Theory[2]

"The Hydrotector measures moisture with a neutron moderation technique. Fast neutrons are emitted from the Americum241/Beryllium source. They pass through the detector without causing a response and also fly into the tested material (generally room structure) bouncing around in the material and gradually slowing down in velocity as a result of the many collisions with the atoms which make up the material or structure.

The detector tubes, (Boron Tri-Fluoride (BF3) detectors), are responsive only to weak, slow, "Thermal" energy neutrons are not at all responsive to the faster velocity neutrons. Thus, the detectors only give a pulse to the electronics when they are struck by

2 Reprinted from the Hydrotector manual.

"Thermal" neutrons which have experienced enough collisions to be slowed down to "Thermal" energy velocity.

Neutrons ricochet strongly from large atoms and approximately 300 collisions are required before the fast neutrons are slowed down to "Thermal" velocity and are visible to the detector tubes.

Neutrons are dramatically slowed down, however, when they collide with hydrogen atoms. Hydrogen atoms are the same size as a neutron and the loss of energy the neutron in colliding with the hydrogen nucleus is very great as opposed to the collisions with the larger atoms commonly found in construction materials and soils. In fact, only 20 collisions are required to "thermalize" a fast neutron as opposed to the 300 collisions with heavier atoms than hydrogen.

The primary source of hydrogen in most materials is in water (H_2O). Thus, the presence of water will produce a substantial "cloud" of thermal neutrons around a fast neutron source (AM241/BE) and the detector will correspondingly produce a substantial electronic pulse output. The more water, the more thermalization can occur; thus, we have a moisture gauge because the signal rate is directly proportional to the quantity of the water present.

It should be apparent, however, that any other source of hydrogen other than water will also produce moderation of the fast neutrons with resultant moisture error. Unfortunately, this is exactly what happens. It is necessary for the operator to recognize that his moisture gauge is really a precise "Hydrogen Analyzer", measuring moisture only because there is no other source of hydrogen other than water present.

Other sources of hydrogen can be organic matter in soil, roots, chemicals, water of hydration (bound water), and the organic material in construction materials such as wood and asphalt in room construction. These other sources of hydrogen are "apparent moisture" in that the Hydrotector is "fooled" into thinking that it is seeing free water as opposed to seeing a combination of hydrogen of both free water and background hydrogen from the other materials.

The background count or signal must be calibrated out with an initial field moisture determination using laboratory measurements or suitable operator judgement techniques."

Thermography Detection Devices

Thermography is the process whereby an Infrared camera records the signature of a radiating body. If the body radiates in a uniform manner then there are little or no differences in the area being scanned. For the purposes of this program, it was thought that fiberglass laminates produced by non-standard methods would give a different 'signature' than those made normally.

	AGA Vision 688	Inframetrics 210
Spectral Range	2 to 5.6 micron	3 to 5 and 8 to 12 micron
Coolant	Liquid N2	Liquid N2
Display	CRT 16 FPS	TV 30 FPS
Temp. Range	-30°C to 2000°C	10°C to 200°C
Thermal Resolution	.2°C	.4°C and .2°C

Test Observations

The observed output from the AGA unit appeared to be superior to that of the Inframetrics. The resolution in the AGA isotherm mode is far superior and resulted in detecting differences that were not otherwise seen. The observations are somewhat less definite using the Inframetrics unit.

The fiberglass reinforced plastic panels (FRP) were positioned vertically, approximately 15 feet from the infrared camera. A uniform heat source was positioned behind the panels and they were warmed to approximately 100 F. The IR camera was then use to scan the panel and the results were displayed on a small television screen. Defects, when present, appeared as shown in Figures 1 and 2.

IR Scanning Analysis of Samples

A Perkin-Elmer model 521 Grating Infrared spectrophotometer was used for this analysis. Sampling procedure was:

A small amount of material was scraped from the sample and mixed with Potassium Bromide (KBR) crystals. This mixture was compressed into wafers about the size of a nickel. This wafer or pellet is aligned in a holder in the spectrophotometer so that light from the IR unit passes through it. The IR unit then passes various wavelengths of light through the sample over a specified IR frequency range. The amount of light passing through the sample at any instant is recorded on a chart recorder. When finished, the chart usually contains many peaks and valleys which indicate the component parts of the sample. A highly trained analyst is needed to accurately interpret the results obtained from this scanning device.

RESEARCH RESULTS

Ultrasonic Inspection of Polymer-Glass Composites

Inspection Procedure

Among the panels presented for inspection were several small sections to be used as "standards". They were presented only as standards with no description of controlled defects or conditions to use for reference. Therefore, these panels were used only as a means of selecting a technique and suitable test frequency for inspecting the remaining panels.

Methods including (1) direct-contact through-transmission, and (2) direct-contact pulse-echo using both single probe and dual-element probes and frequencies of 1.0 MHz, 2.25 MHz, and 5.0 MHz were evaluated. A direct-contact single-probe pulse-echo technique was selected using a 1/2-inch diameter, 5 MHz transducer and a Branson Sonoray inspection system.

5A-C

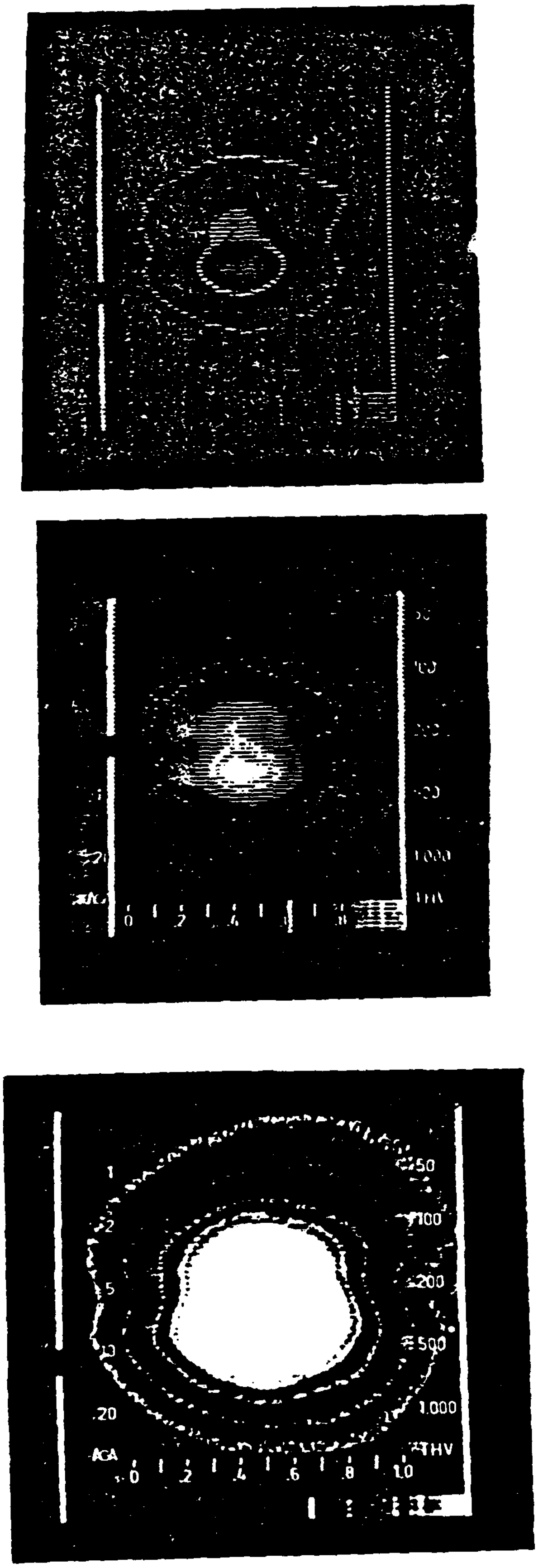
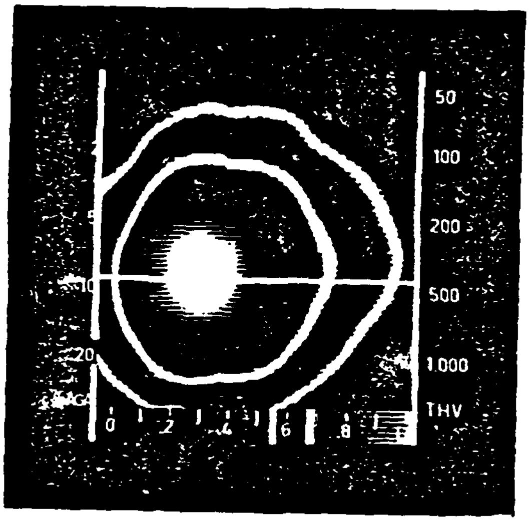
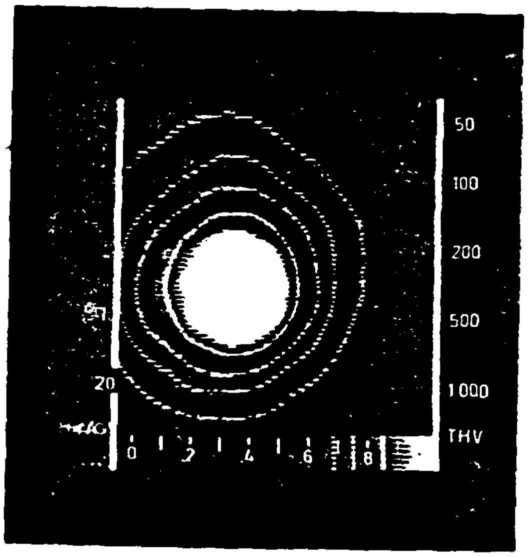


FIGURE 1. PANEL DEFECTS AS DETERMINED BY INFRARED CAMERA

4A-C



4B-C



D-B

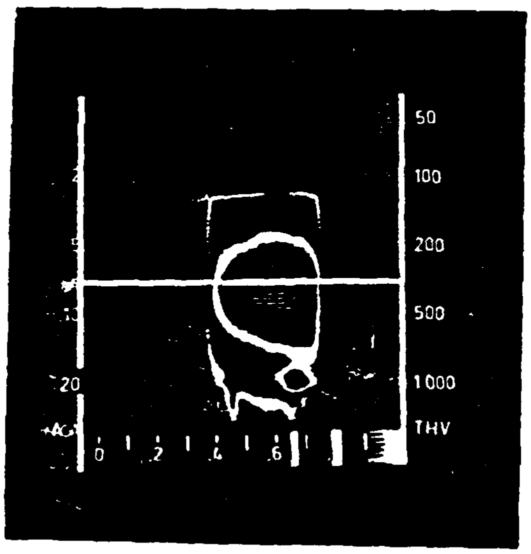


FIGURE 2. PANEL DEFECTS AS DETERMINED BY INFRARED CAMERA

The inspection procedure was based upon the assumption that both the panels and the inspection technique itself were being evaluated. The procedure was planned to minimize cost of inspection probably at the expense of thoroughness in defining defect types and their seriousness.

Inspection was performed by passing a transducer along a thin oil path laid on the smooth surface of each panel. Internal reflections including size, shape and position as well as number were monitored simultaneously. These data provided the information upon which to evaluate the conditions within the panels.

The 5 MHz probe yielded generally excellent penetration through the panels with one exception to be discussed later (see page 14). Complete penetration was verified by occasionally loading the backside of the panel under test with a moistened finger and observing the effect on the echo from the back surface.

The inspection procedure was as follows:

> A parallel grid pattern of lines approximately 2-1/2 to 3 inches apart was laid out by the thin oil lines. A light oil was used to couple the transducer to the panel. Positions along the grid lines representing poor to no through transmission were identified by graphite pencil marks. These marks are easily wiped or washed off the surface. Notes regarding observations were also recorded as the inspection proceeded. As the inspection progressed, interpretation of the signals on the oscilloscope screen became easier and the speed of inspection increased.

The results of the inspection are summarized in the following section.

Inspection Results

Panel A Circle D. This panel was scanned along parallel lines in one direction only. There was generally no reflection from the back-surface, indicative of poor cure or resin penetration.

Panel B Circle D. This panel produced generally acceptable transmission properties with occasional indications of internal discontinuities such as delamination, differences in curing of polymer, poor fiber bond, or similar. No defect indications were marked on this panel.

All of the following panels were inspected in the order presented.

Panel C Circle D. The inspection revealed:

(1) Good transmission through the complete thickness in some areas indicating good distribution and curing of polymer materials.

(2) Some areas allowed poor echoes from the back surface indicating considerable scattering or spotty curing or bonding. The panel was very heterogenous.

(3) In some areas, good internal reflections were obtained which were not reflected from the back-surface. These are indicative of internal delamination. No deflect areas were marked on this panel.

Panel D Circle D. Fair to excellent back echo over some areas of the panel. High damping in plate associated with fair back-echo. Occasional indication of delamination and in some areas there was poor through transmission.

Panel E Circle D. Distances along each grid line corresponding to no transmission to the back-surface were identified by pencil mark. The received echoes were typical of delaminations or poor bonds between layers, lack of complete resin penetration, or similar reflection or scattering conditions.

Panel F Circle D. In areas close to two adjacent sides, transmission was poor to spotty as marked on the panel, indicative of delamination or poor resin distribution. Transmission through the center and along one edge was generally good as indicated by good back-echo.

Panel 1 Circle A. This panel provided excellent back-echoes along all lines, indicative of materials of good quality.

Panel 2 Circle A. This panel allowed generally good indications

Panel 2 Circle A. This panel allowed generally good indications with the exception of two short narrow strips near one edge. These strips may be delaminations brought about in sawing the panel.

Panel 3 Circle A. This panel provided excellent back-echoes along all lines, indicative of materials of good quality.

Panel 4 Circle A. Echo patterns were consistently good to excellent along each line checked.

Panel 5 Circle A. Transmission is variable and shows considerable scatter. Some areas indicate possible delamination. Occasional fair to good back echo. A considerable area shows loading which means that energy goes into the panel and is lost by scattering or other attenuation mechanisms. Some areas indicate no through transmission. For most of the panel, indications are not highly damped which means that there may be delaminations or discontinuities.

Panel A Circle B. This panel provided excellent back-echoes along all lines, indicative of materials of good quality.

Panel B Circle B. This panel provided excellent back-echoes along all lines, indicative of materials of good quality.

Panel C Circle B. This panel provided excellent back-echoes along all lines, indicative of materials of good quality.

Panel D Circle B. The texture of this panel differs from that of the other panels. No back-echoes or reverberations were seen in this panel using the 5 MHz probe. From the appearance of the indications on the oscilloscope screen and the visual appearance of the board design, one might conclude that the texture causes excessive scattering at 5.0 MHz and that a lower frequency might be necessary for ultrasonic inspection. The indications may also be attributed to

(1) Lack of good polymer distribution or bonding to fibers

(2) Delamination, although no signals were observed that are characteristic of delaminations.

Panel E Circle B. Ultrasonic indications from this panel were generally good with the exception of some areas identified by pencil mark which gave indications similar to those of delaminations.

Panel 1A Circle C. Conditions in this panel appear to be generally good along the lines inspected.

Panel 2A Circle C. Conditions in this panel appear to be generally good along the lines inspected.

Panel 3A Circle C. Conditions in this panel appear to be generally good along the lines inspected.

Panel 4A Circle C. Signals from back surface were generally good to excellent in this panel. May contain some delamination, but, without better definition of a reject, this panel rates as good.

Panel 5A Circle C. With the exception of two short distances along one edge and one short distance and one corner where indications were typical of delaminations,this panel produced good to excellent transmission and reverberation signals. This panel appears to be of generally good quality.

Inspection Matrix

During a review meeting at Battelle Columbus Division on February 6, 1987, a matrix of NDT capabilities relative to fiberglass panels was suggested.

This matrix as it relates to ultrasonic testing of fiberglass panels would appear as follows:

Condition	Probable Ultrasonic Response
Undercure	
a. Resin path continuous and good bond to fibers.	Back echo received accompanied by damping and little scatter. Reduced velocity of sound.
b. Poor bond, porosity, etc.	Poor to no back echo. Attenuation both by scattering and absorbtion (damping). Reduced velocity of sound.
Overcure	
a. Resin path continuous and good bond to fibers.	High back echo. Decreased velocity of sound.
b. Poor bond, porosity, etc.	Poor to no back echo. Attenuation due to scattering. Velocity of sound similar to a to slightly lower.
Normal	Good back echo. Attenuation and velocity of sound depends upon definition of optimum conditions.
Wet	Depends upon reaction of moisture with the resin. Would expect poor bonding to fibers, porosity due to water vapor, and probably an effect on quality of resin cure. These conditions could cause high acoustic scattering, possibly high damping(especially if quality of resin is affected) and poor acoustic transmission. Velocity of sound might be reduced.
Cold	Poor lamination would degrade the ability to transmit through the panels.
	Additional Defects
Delamination	Interrupts transmission. May give good reverberations if no additional defects are encountered as above.
Porosity	Causes severe scattering and echoes depending upon spacing and geometry of clusters.

The above matrix summarizes the results of our preliminary investigation. The matrix will require verification.

Discussion of Results

The results presented in this report obviously are qualitative rather than quantitative. More thorough inspection requires the use of standards containing known defects and an optimization of frequency selection and inspection mode.

Regarding the comment that degree of cure of the polymer may be a factor in some panels, the probability that this is the case depends upon the nature of the polymer during the fabrication of the panels. The more likely case with material formed from fibers and fluid polymer to be cured later is poor bonding and penetration into the fiber bundles. If the fibers are well wetted with polymer and the path from surface to surface is continuous, back-echoes will be observed. Velocity of sound changes with degree of cure so that position of the back echo will be affected.

The position of the back-echoes appeared to be similar for all panels of a given thickness. This indicates that velocity of sound apparently remained constant throughout each panel so that degree of cure may not have been a factor in any of the panels.

Our observations were primarily of ability of the ultrasonic energy to penetrate the panels, scattering, and the appearance of the echo patterns.

Recommendations

We believe that this study shows that the fiberglass boat hulls can be inspected ultrasonically. To develop a good inspection system it will be necessary to:

(1) Develop a standard for each panel construction including defect types, etc.

(2) Design or develop a system including transducers and instrumentation for processing data automatically as the hull is scanned.

(3) Design an immersion inspection system as a practical approach since the materials are for use in boat hulls.

Inspection of Polymer-Glass Composites by Dielectric Methods

This measuring unit was obtained from Sovereign Chemical Industries Limited and measures changes in capacitance of materials over an area rather than between two points. Measuring the area reduces the variations that are present in most materials because it averages out large differences that were sometimes found using the two pin method. This unit is described as a moisture detector but it should be capable of measuring any change in the dielectric of a material if the material is not uniform and the dielectric constants of the materials under test are different. For example, if unreacted curing agent were present, a test reading should reveal a different constant (indicated by a different meter reading). However, it is unknown as to what to expect from a fiberglass layup that was fabricated using unacceptably low layup temperatures.

This instrument was also used to measure several sailboats (stored on land for the winter) at a local marina. The above waterline readings were used as a baseline and were taken to indicate no moisture present. Blisters were present on many of these boats and the instrument recorded many variations over the blistered areas. All of the readings were higher over and near blisters than those recorded above the waterline. Thicker areas of the ship bottom resulted in different readings than normal and any metal plates imbedded in the bottom resulted in very high readings. On boats with no history of blistering the below waterline readings were only slightly higher than those taken from above the waterline.

Inspection Procedure

Two standard panels, a blue fiberglass orthophthalic resin hand layup and a white fiberglass isophthalic resin hand layup, were used as controls. Six readings were taken on each panel and then averaged. No differences were noted between the two standards. All panels were evaluated by placing them on the same substrate as the standard panels while the measurements were made. This procedure precludes the possibility that erroneous readings could be obtained due to substrate thickness or composition. The most sensitive scale on the instrument was utilized for this work because it gave the widest range of readings. Twenty-one experimental panels were evaluated and four of these were prepared having excess water on the fiberglass cloth during layup.

Table 1 lists the results of this inspection. These results indicate higher moisture content than the standard in only two panels. The moisture content of one panel was lower than that of the standards. Since the instrument is very sensitive to moisture it was concluded that the "excess moisture" panels may not have been prepared uniformly.

Inspection of Polymer-Glass Composites by Neutron Bombardment

The device Hydrotector is a hydrogen detector which emits fast neutrons that penetrate the substrate under test. Fast neutrons decrease slightly in velocity when they strike any atom but slow dramatically when they collide with a hydrogen atom. When their velocity has decreased to a certain point, a detector in the instrument counts these slow neutrons and this determines the hydrogen content (correlated to the amount of moisture present) once a baseline has been established. This instrument was suggested as one means of detecting moisture in fiberglass boat hulls. Some preliminary work was done by the company that designed this unit (CPN Corp.) and this work indicated that the detector in this unit would indeed detect moisture in fiberglass boat hulls.

Inspection Procedures

Twenty-one experimental panels were inspected with this instrument. Two controls were used as before. Ten readings were taken on each panel and then averaged. Each reading consisted of approximately 10 bursts of neutrons into the sample. The higher the number of the average reading of the sample, the higher the moisture content. Table 2 lists the results of this study.

TABLE 1. TEST RESULTS ON FIBERGLASS PANELS USING THE SOVEREIGN MOISTURE METER[a]

Panel Number	Test Readings						Average
Blue Fiberglas (control)	10	10	10	10	10	10	10
White Fiberglas (Control)	10	10	10	10	10	10	10
A circle B	10	10	10	10	10	10	10
B circle B	10	10	10	10	10	10	10
C circle B	10	10	10	10	10	10	10
D circle B	47	43	48	54	60	57	51.5
E circle B	10	10	10	10	10	10	10
1 circle A	10	10	10	10	10	10	10
2 circle A	10	10	10	10	10	10	10
3 circle A	10	10	10	10	10	10	10
4 circle A	10	10	10	10	10	10	10

TABLE 1. (Continued)

Panel Number	Test Readings						Average
5 circle A	25	25	25	25	25	25	25
1B circle C	10	10	10	10	10	10	10
2B circle C	10	10	10	10	10	10	10
3B circle C	5	5	5	5	5	5	5
4B circle C	10	10	10	10	10	10	10
5B circle C	10	10	10	10	10	10	10
A circle D	10	10	10	10	10	10	10
B circle D	10	10	10	10	10	10	10
C circle D	10	10	10	10	10	10	10
D circle D	10	10	10	10	10	10	10
E circle D	10	10	10	10	10	10	10
F circle D	10	10	10	10	10	10	10

(a) All data can be found in Laboratory Record Book No. 42717.

TABLE 2. TEST RESULTS USING THE HYDROTECTOR®

Panel Identification	Count Values										Total field count	Average field count	Field count /standard count = Ratio	Moisture content = Ratio x A + (-B) (a)
Standard Count	5690 5686	5808 5694	5802 5814	5640 5744	5958 5810	total = 57646 average standard count = 5764								
Blue Fiberglas (control)	764	718	760	674	756	786	746	710	770	716	7340	734	0.127	0.807 lbs cu/ft
White Fiberglas (control)	734	796	864	788	798	831	830	840	846	838	8165	816	0.142	1.226 lbs cu/ft
A circle B	792	796	728	760	732	784	780	698	756	722	7548	754	0.131	0.989 lbs cu/ft
B circle B	736	748	754	800	746	736	712	770	770	730	7510	751	0.13	0.894 lbs cu/ft
C circle B	740	760	782	776	862	724	738	772	802	660	7536	753	0.131	0.904 lbs cu/ft
D circle B	744	726	720	756	734	720	710	714	750	786	7368	736	0.128	0.817 lbs cu/ft
E circle B	793	724	790	794	732	754	810	776	720	800	7693	769	0.133	0.986 lbs cu/ft
1B circle C	814	832	820	802	854	770	846	852	800	816	8206	820	0.142	1.246 lbs cu/ft
2B circle C	850	844	800	784	834	842	850	840	836	832	8320	832	0.144	1.307 lbs cu/ft

TABLE 2. (Continued)

Panel Identification	Count Values										Total field count	Average field count	Field count /standard count = Ratio	Moisture content = Ratio x A + (-B) (a)
Standard Count	5698 5686	5800 5694	5802 5814	5640 5744	5958 5810	total = 57646 average standard count = 5764								
Blue Fiberglas (control)	764	718	700	674	756	786	746	710	770	716	7340	734	0.127	0.807 lbs cu/ft
White Fiberglas (control)	734	796	864	788	798	831	830	840	846	838	8165	816	0.142	1.228 lbs cu/ft
3B circle C	834	740	786	790	768	826	778	822	824	840	8008	800	0.139	1.144 lbs cu/ft
4B circle C	810	790	852	838	826	758	790	822	774	810	8070	807	0.14	1.18 lbs cu/ft
5B circle C	766	796	804	862	820	810	870	770	830	800	8128	812	0.141	1.206 lbs cu/ft
A circle D	788	800	780	720	716	774	730	760	748	758	7574	757	0.131	0.924 lbs cu/ft
B circle D	758	728	728	732	728	738	688	730	750	744	7324	732	0.127	0.797 lbs cu/ft
C circle D	746	758	726	712	734	726	660	750	762	704	7278	727	0.126	0.771 lbs cu/ft
D circle D	700	690	644	686	680	694	714	694	690	744	6936	693	0.12	0.598 lbs cu/ft

TABLE 2. (Continued)

Panel Identification	Count Values										Total field count	Average field count	Field count /standard count = Ratio	Moisture content = Ratio x A + (-B) (a)
Standard Count	5690 5688	5808 5694	5802 5814	5640 5744	5958 5810	total = 57640 average standard count = 5764								
Blue Fiberglas (control)	764	718	780	674	758	786	746	710	770	716	7340	734	0.127	0.807 lbs cu/ft
White Fiberglas (control)	734	796	864	788	796	831	830	840	846	838	8165	816	0.142	1.226 lbs cu/ft
E circle D	638	684	646	678	656	718	726	700	744	630	6796	679	0.118	0.526 lbs cu/ft
F circle D	670	672	680	736	674	636	764	742	660	730	6964	696	0.121	0.613 lbs cu/ft
#1 circle A	756	852	852	868	768	796	806	812	806	716	8034	803	0.139	1.159 lbs cu/ft
#2 circle A	874	738	772	832	842	818	794	772	834	750	8018	801	0.139	1.149 lbs cu/ft
#3 circle A	862	818	768	874	850	866	760	778	770	782	8128	812	0.141	1.205 lbs cu/ft
#4 circle A	810	794	690	722	690	762	764	740	820	802	7594	759	0.132	0.935 lbs cu/ft
#5 circle A	870	842	830	840	800	830	848	820	782	850	8312	831	0.144	1.302 lbs cu/ft

(a) A=29.42294, B=-2.93975. These values are based on the Standard Count.

The instrument is first calibrated against a standard panel following the instructions received with the instrument. This value is termed the standard count. The experimental panels are then carefully tested using the detector and by recording each reading on the instrument. This is called the field count. After averaging ten field counts per panel, the field count is divided by the standard count and this is called the Ratio. The moisture content is then determined by multiplying the Ratio by A and adding (-B) to it. A and B were obtained by extrapolating the values from previous standard counts furnished with the instrument.

The results shown in Table 2 are difficult to interpret because they do not indicate excess moisture in panels known to have excess moisture. The variation in readings of panels from each set are not great. For example, in series circle B, the variation is from .8 to 1.0 lb of water per cubic foot. The variation is greater in the two standard panels. It was concluded that this instrument may have been designed to accurately measure moisture in thick roofing materials, but may need some modification to accurately measure thin (1/4") panels.

Inspection of Polymer-Glass Composites by Thermography

Thermography's most familiar use is in the detection of heat loss from homes or businesses. Not so familiar is its use commercially to detect overheating in machinery parts and detection of flaws in various components. It works by recording infra-red radiation emitted by a subject. If the subject material is not uniform then the radiation will not be emitted in a uniform manner. The fiberglass panels being evaluated should be uniform unless they contain flaws. If they do contain flaws then it is possible that a sensitive thermography detection camera could pick up these imperfections. A thermographic inspection analysis was made on the fiberglass reinforced plastic panels furnished by the Coast Guard.

Inspection Procedure

The results of the observations are shown in Table 3. Various flaws in the fiberglass panels were identified using this procedure but the

TABLE 3. DEFECT VISIBILITY USING THE AGA VISION 680 CAMERA

Panel Identification	Defect Visibility High	Medium	Low	None
A circle D		X		
B circle D				X
C circle D				X
D circle D			X	X
E circle D				X
F circle D				X
1 circle A				X
2 circle A				X
3 circle A				X
4 circle A				X
5 circle A			X	X
1A circle C			X	
2A circle C		X	X	
3A circle C			X	
4A circle C			X	
5A circle C	X			
1B circle C			X	X
2B circle C			X	X
3B circle C		X		
4B circle C			X	X
5B circle C		X	X	
A circle B			X	X
C circle B			X	X
D circle B			X	X
Blue Standard			X	
White Standard			X	

cause of the flaws is unknown at this time. To be effective, a flaw must be related to a specific defect in the panel and at the time of the inspection this information was not available. The flaws that were detected and marked medium in Table 3 were very visible using Inframetrics isotherm mode and even more so with the AGA. The flaws that were detected and marked low were readily apparent using the AGA unit. They were not as pronounced using the Inframetrics unit.

The thermal uniformity of the heat source is of great importance in the detection process. Every attempt was made to reduce any influence on the observations that could be attributed to the thermal source. It was difficult to obtain meaningful information from these panels because their small size made them difficult to heat properly.

Figures 1 and 2 show AGA photographs of four panels. These are 5A circle C, 4A circle C, 4B circle C, and D circle B. The three photos of 5A circle C show a flaw that looks like a figure 8 display. The 4A circle C photo is more uniform but something in the panel disturbed the thermal pulse. The 4B circle C photo in nearly uniform but the thermal pulse is still disturbed. The photo of panel D circle B is an example of a thermal pulse from a smaller panel. It is difficult to determine if any flaws exist due to its small size.

Chemical Analysis of Polyester-Fiberglass Panels

Infrared analysis of the laminate panels was initially undertaken to determine which type of resin was used in constructing the panels by the various boat manufacturers and resin suppliers. Resin samples were scraped from the gel coat side (smooth finish) and the glassy side (rough side) of each panel and made into a standard KBr pellet for analysis by infrared (IR) sprectroscopy. Table 4 below indicates which resin types were found in the various panels.

While the particular resins have been identified in each panel, the panels are not similar in aliphatic ester (i.e., nonring containing ester) or in the ratio of various reactive species within the laminate. Differences exist in ratios of styrene to ester and ester to ester ratio for most of the sample panels. While differences in these ratios cannot be compared between

TABLE 4. TYPE OF POLYESTER PRESENT IN BOAT PANELS[a]

Sample	Gel Coat	Glass
Blue Standard	Orthophthalic	Orthophthalic
White Standard	Orthophthalic	Isophthalic
Circle A Series	Isophthalic	Ortho
Circle B Series	Isophthalic	Ortho
Circle C Series	Isophthalic	Ortho
Circle D Series	Orthophthalic	Ortho

(a) As measured by IR.

sets of sample panels because of the slightly different chemistry involved, differences within a series can be postulated to relate to differences in cure. Table 5 lists some of the facts ascertained concerning ester and styrene ratios for the panels from the two different sides.

While all samples contained a small amount of styrene on the glassy side, the interference caused by the filler and the low concentration of styrene effectively prohibit any accurate estimate of this ratio.

Based on the IR data presented it is difficult, if not impossible, to predict which of the panels belong in the cells of the cure matrix (i.e., Normal Cure, Overcure, Undercure, Wet Cure, or Cold Cure). However, it is expected that the Undercure, Wet Cure and Cold Cure would be similar in nature since each of these panels would be expected to be undercured to some degree. Since the styrene content could be related to residual monomer at the surface, then the higher the styrene:ester ratio, the more likely it is that panels are undercured. Overcured panels would then be expected to have a lower styrene:ester ratio. Using this unsubstantiated hypothesis then, the panels in each series can be lumped into three groups: Normal, Undercure or Overcured. This has tentatively been done in Table 6.

IR is useful in quickly determining the composition of the various resin layers. However, much more study would be necessary to develop a reproducible technique for determining cure of the materials. Many IR spectra peaks other than the styrene:ester ratio could possibly be used to help identify properties of interest. In general, this would involve considerable more funds than available at this time.

CONCLUSIONS

It is apparent that to develop a satisfactory technique to predict boat hull blistering it will requireadditional effort (both time and money). It is also apparent from the work that has been done that an ultrasonic technique may be the best way to approach a solution to the problem.

TABLE 5. COMPARISON OF CHEMICAL SPECIES WITH EACH PANEL

Series	Gel Coat Styrene/CH_2 Ratio	Glassy Side
White Standard	.67	Isophthalate based
Blue Standard	.42	Orthophthalic based
<u>Circle A Series</u>		
1	.58	Major aliphatic ester; ratio of
2	.55	1 part phthalic to 2-3 parts
3	.54	maleic ester likely
4	.57	
5	.56	
<u>Circle B Series</u>		
A	.58	Very similar to Blue Standard
B	.58	with aromatic to aliphatic com-
C	.58	position: ~2:1 phthalic: maleic
D	.60	
E	.57	
<u>Circle C Series</u>		
1A	.72	Very similar to A in composition
2A	.65	of esters, e.g., 1 part phthalic:
3A	.73	2-3 parts maleic
4A	.69	
5A	.70	
<u>Circle D Series</u>		
A	1.02	Estimate of 2 parts phthalic per
B	1.11	1.5 parts maleic for composition
C	1.18	
D	1.11	
E	1.15	
F	1.13	

TABLE 6. PREDICTION ON CURE OF PANELS

Series	Undercure	Normal	Overcure
Circle A	1,4,5	2	3
Circle B	4,5?	?	?
Circle C	1,3,5	4	2
Circle D	3,5,6	2,4	1

After the completion of the evaluation of the fiberglass panels by the various means described in this report, tables were prepared using the data obtained. The data was then compared against the panel's composition which was supplied to us at the end of the program by the U. S. Coast Guard. These results are shown in four tables, 7 through 10. Numerical values are shown where possible and subjective values are shown elsewhere.

From the work performed using the various instruments to detect premature failures in fiberglass laminates, it appears that two of the detecting units used in this research hold some promise for evaluating boat hulls.

Moisture Meter

The first of these, the Sovereign Moisture Master, will detect moisture in fiberglass boat hull laminates. Tables 7 through 10 indicate that high readings were obtained (indicative of excess moisture) on two of the four 'wet' panels prepared for use in this test. The panels from the participating companies were probably not prepared with uniformly 'wet' fiberglass mat so it is not known what minimum moisture content is necessary in the fiberglass to obtain a moisture reading. Subsquently, this instrument was also used on blistered and unblistered boat hulls that were stored on land for the Winter. The hull area above the waterline was used as a reference surface for these tests. All the readings near the blistered areas on the boat hulls produced readings higher then the reference values. Other boat hulls examined that did not show blistering did not produce values higher than reference readings. More tests need to be run using fiberglass laminates with known percentages of water to determine if this instrument can be useful in plant analysis or whether it is only useful on blistered boat hulls. The data obtained from these tests should be correlated with some other test, possibly the hot water exposure test now under way at The University of Rhode Island, to determine if moisture detection can be related to actual blistering.

TABLE 7. FIBERGLASS PANEL SERIES-CIRCLE A[a]

Sample No.(1)	Sovereign Moisture Meter(2)	Hydro-tector (3)	Ultra-sonic probe (4)	Thermo-graphy (5)	Infra-Red Analysis (gelcoat/back) (6)	Panel Ident-ity(7)
#1 circle A	10	1.3	good	none	iso / ortho	Std.
#2 circle A	10	1.2	good	none	iso / ortho	Cold Cure
#3 circle A	10	1.3	good	none	iso / ortho	Low Cat.
#4 circle A	10	1.0	good	none	iso / ortho	Excess Cat.
#5 circle A	25	1.4	poor	low	iso / ortho	Water Added

(a) Footnotes for Tables 7 through 10 are found on page 33.

TABLE 8. FIBERGLASS PANEL SERIES-CIRCLE B

Sample No.(1)	Sovereign Moisture Meter(2)	Hydro-tector (3)	Ultra-sonic probe (4)	Thermo-graphy (5)	Infra-Red Analysis (gelcoat/back) (6)	Panel Ident-ity(7)
A circle B	10	1.0	good	low	iso / ortho	Std.
B circle B	10	.9	good	low	iso / ortho	Excess Cat.
C circle B	10	1.0	good	low	iso / ortho	Low Cat.
D circle B	51.5	.9	poor	low	iso / ortho	Water Added
E circle B	10	1.1	fair	low	iso / ortho	Cold Cure

TABLE 9. FIBERGLASS PANEL SERIES-CIRCLE C

Sample No.(1)	Sovereign Moisture Meter(2)	Hydro-tector (3)	Ultra-sonic probe (4)	Thermo-graphy (5)	Infra-Red Analysis (gelcoat/back) (6)	Panel Ident-ity(7)
1A circle C	10	1.3	good	low	iso / ortho	Std.
2A circle C	10	1.4	good	low	iso / ortho	Excess Cat.
3A circle C	5	1.2	good	low	iso / ortho	Low Cat.
4A circle C	10	1.3	good	low	iso / ortho	Water Added
5A circle C	10	1.3	good	medium	iso / ortho	Cold Cure

TABLE 10. FIBERGLASS PANEL SERIES-CIRCLE D

Sample No.(1)	Sovereign Moisture Meter(2)	Hydro-tector (3)	Ultra-sonic probe (4)	Thermo-graphy (5)	Infra-Red Analysis (gelcoat/back) (6)	Panel Ident-ity(7)
A circle D	10	1.0	poor	low	ortho / ortho	Std.
B circle D	10	.9	good	low	ortho / ortho	Excess Cat.
C circle D	10	.8	fair	low	ortho / ortho	Low Cat.
D circle D	10	.7	fair	medium	ortho / ortho	Water Added
E circle D	10	.6	fair	low	ortho / ortho	Cold Cure
F circle D	10	.6	fair	low	ortho / ortho	*

* Chopped fiberglass used next to gel coat instead of mat. Otherwise standard procedure.

Footnotes for Tables 7 through 10

(1) The sample number was furnished by the manufacturer of the panels. The letter in parentheses was placed on the panel by Mr. Don Ellison of the Coast Guard as his indification.

(2) This is a radio frequency probe. A value of 10 on the meter scale appears to be a base line reading on standard fiberglass panels. Higher values would indicate above normal moisture.

(3) The probe uses a neutron moderation technique to determine moisture. The values given are in pounds of water per cubic foot of fiberglass laminate.

(4) This unit uses high frequency sound waves to determine the structure of the laminate. The values, good, fair, or poor are indicative of the reading obtained from evaluating the panels. A normal (good) panel would echo most of the sound waves impressed on it. A panel rated fair would show some scattering of wave and a panel rated poor would indicate scattering as well as dampening.

(5) This instrument measures heat radiation from an above ambient temperature object. Defects in the laminates are recorded as uneven radiation patterns. Low and moderate readings indicate some defect in the panel. It was not possible to identify the defect without further evaluation.

(6) This column identifies the gel coat and laminate structure of the panel as to whether the polyester is based on isophthalic esters or orthophthalic esters.

(7) Panel Identity was unknown to Researchers until all tests were run. This was done to reduce bias.

Ultrasonic Detection

The second test method which holds promise, ultrasonic detection, appears to be able to 'probe' inside a fiberglass layup and detect any number of anomalies that may be present (See Tables 7 through 10). One of the problems with the ultrasonic test method is that there are too many 'defects' that the instrument senses and it is difficult to sort out the good and bad readings without prior knowledge of which defects are important and which ones are not. Since this was an exploratory program, only limited time and effort could be spent on any one technique. What is needed now is to determine what defects are important (i.e.,cold cure, excess catalyst, etc.) so that a technique can be developed that will differentiate between the most important and minor (unimportant) faults. Appendix A contains excerpts from a book relating to the examination of fiberglass laminates using ultrasonics.

Thermography is another technique that could be developed to analyze and predict failures in fiberglass boat hulls but it is not believed to be as sensitive to aberrations as the ultrasonic technique. Tables 7 through 10 list the results obtained from this procedure. This method would be easy to use once standardized but it is believed that it would be more difficult than ultrasonics to implement. Therefore, it is not recommended as a possible tool for any future development work.

The Hydrotector instrument is believed to have been designed to analyze material thicknesses greater than those examined on this project. No success was had in trying to analyze any of the plastic laminates used in this program. It is not recommended for any future work.

The use of Infrared Spectroscopy by itself would not be valuable in predicting fiberglass failures but in conjunction with other techniques it would provide some degree of confidence initially that one of the other techniques was being used to evaluate the boat hulls correctly. Again, more time must be invested in this technique to prove its feasibility.

RECOMMENDATIONS

It would be appropriate to compare data recorded here with hot water test data conducted on these panels at the University of Rhode Island. This may offer a correlation of onset of blistering with fiberglass composition. This information could prove to be valuable in any future work sponsered by the U. S. Coast Guard or by boat manufacturers or resin suppliers. More fiberglass panels need to be prepared with precise measurements of the various components so standards can be developed that can be examined at various wavelengths and assorted frequencies. Special transducer heads must be developed that can scan a boat hull quickly and thoroughly. Standard patterns for each failure mode could be used in conjunction with ultrasonic signatures to either signify a pass or fail mode for each hull being scanned. This research work would be costly and may take one to two years to complete. The funds for such a program could come from a multiclient approach that would include a number of boat and resin companies sharing the costs as well as the research results. The boat hull blister problem does not seem to be diminishing and all responsible boat manufacturers should be willing to invest in a program that will minimize their problems.

APPENDIX A

APPENDIX

The following excerpts are pertinent to the present study on the inspection of fiberglass panels.

1. From D. Ensminger, Ultrasonics, Marcel Dekker, Inc., New York (1973)
pp. 346-347.

IX. FIBROUS-BONDED COMPOSITES

A heterogeneous distribution of elastic properties is characteristic of certain material such as graphite-fiber materials, filament-wound structures, and epoxy-glass laminates. Variations in the degree of cure or in the distribution of the binding agent lead to variations in the velocity of sound and in the attenuation of sound by scattering. The strength of the bond between adjacent fibers also affects the velocity and scattering. Ultrasonic measurements of thickness of reasonable accuracy may not be possible without the aid of a secondary method since such measurements are based upon velocity of sound. Resolution and defect sensitivity also are affected. However, degree of cure and quality of bonds are important and their influence on the propagation of ultrasonic waves makes ultrasonics of considerable value in the nondestructive inspection of composite materials. Measurement of the velocity of sound generally is a good method of determining variations in the strength of composite materials. Attenuation can be correlated with delaminations, porosity, and resin content. Defects can be located by through-transmission or pulse-echo methods.

2. From J. Krautkramer, etal, Ultrasonic Testing of Materials, 3rd ed. (1983) p. 552.

In recent years the problem has come up concerning quality tests on glass-fibre reinforced and carbon-fibre reinforced polymers by means of ultrasonics. Specific defects which have a detrimental effect on the properties of GFRP and CFRP are irregular laminar structure, badly cured spots, and separations between layers. Larger defects can be detected satisfactorily by the through-transmission method and by beaming normal to the surface at testing frequencies of usually 1 to 2 MHz. Uniformity in structure and curing also can be evaluated by measuring the acoustic velocity, although this value is influenced also by harmless porosities in the resins. Information concerning the distribution of the glass fibres in the layers can be obtained by means of two angle probes coupled to the same surface of the material concerned which are directed at each other, by determining the transit time of the through transmission (Schaper and Stelling) [640,641]. Further bibliography: [277-552]).